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1 557 225

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(19)

(54) SULFINYL COMPOUNDS AND PROCESSES FOR PREPARING SAME

(71) We, KAO SOAP COMPANY LIMITED, a Japanese Company, of 1,1-chome, Nihonbashi-Kayabacho, Chuo-ku, Tokyo, Japan, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to novel compounds which have an antibiotic function. More specifically, the present invention relates to derivatives of acrylic acid, which derivatives have a sulfinyl group at the β-position of the acid.

Many problems arise when conventional antibiotic chemicals are used. A primary problem is that each of the known antibiotic chemicals can only be applied to a small group of systems or species of micro-organisms. Under the present circumstances, it is therefore necessary to subject a number of available antibiotic chemicals to various tests in order to select the specific chemical which is suited for application to the particular system or species of micro-organisms. Although antibiotic chemicals of the phenol system have been widely used, this kind of chemical, in general, has only a narrow spectrum of antibiotic activity and it must be used at a high concentration. Antibiotic chemicals of the halogen-substituted aromatic compound system, which are another kind of widely used antibiotic chemical, tend to accumulate in the natural world without being decomposed, which causes another kind of problem. It is also known that invert soaps exert remarkable antibiotic activities at low concentrations. However, it is difficult to apply invert soaps to a system in which it is desired to avoid lathering of the soaps or to an anionic emulsion system, because the invert soaps form insoluble complexes with such systems.

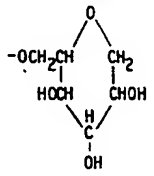
The present invention has been developed as a result of our vigorous efforts to solve the aforementioned problems.

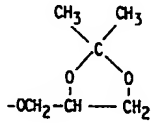
The object of the present invention is to provide compounds which have an antibiotic function and which can be widely applied to various uses.

The present invention provides compounds having the following formula (1):

$$RSO—CH=CHX \qquad (1)$$

In the above formula, R is alkyl or alkenyl having 1 to 20 carbon atoms, or aryl such as aryl having 6 to 10 carbon atoms; and X is —COY, wherein Y is (1) $—O(CH_2CH_2O)_mH$, wherein m is zero or an integer from 1 to 12, or (2) —OM, wherein M is an alkali metal, an alkaline earth metal or NH_4, or (3) $—O(CH_2CH_2O)_mR_1$, wherein m is as defined above and R_1 is alkyl having 1 to 20 carbon atoms, or (4) a hydroxyl-substituted alkoxy group obtained by removing one hydrogen atom from one hydroxyl group of a polyhydric aliphatic alochol or





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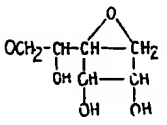
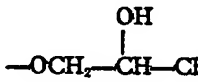
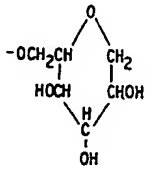


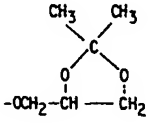
or (5) —NR'R", wherein R' is selected from hydrogen, alkyl having 1 to 20 carbon atoms, and hydroxyalkyl having 2 to 6 carbon atoms, and R" is selected from hydrogen, alkyl having 1 to 20 carbon atoms, and substituted alkyl having 2 to 6 carbon atoms wherein the substituent is selected from hydroxyl and a sulfo group in the form of a salt (—SO_3M_1, wherein M_1 is an alkali metal).

Preferably R is a straight chain alkyl or alkenyl group having 3 to 18 carbon atoms; Y is preferably selected from alkoxy having 1 to 3 carbon atoms, alkoxyethoxy having 1 to 3 carbon atoms in the alkyl moiety —$O(CH_2CH_2O)_2R'$ where R' is alkyl having 1 to 3 carbon atoms,





—$O(CH_2CH_2O)_mH$ (m=1 to 12),



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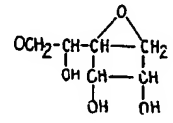


and where Y is —NR'R", R' is preferably selected from hydrogen, alkyl having 1 to 3 carbon atoms, and hydroxyalkyl group having 2 or 3 carbon atoms; and R" is selected from hydrogen, alkyl having 1 to 3 carbon atoms, hydroxyalkyl having 2 or 3 carbon atoms, and substituted alkyl group having 2 or 3 carbon atoms and wherein the substituent is —SO_3M wherein M is an alkali metal.

The compounds having the above formula (1) can be obtained by oxidizing compounds having the formula (2) with an inorganic or organic peroxide.

$$RS—CH=CHX \qquad (2)$$

wherein R and X in the formula (2) are the same as defined hereinabove with reference to the formula (1).

The formula (2) starting compounds and their preparation are disclosed and claimed in British Patent Application No. 26353/76 (Serial No. 1 528 853).

Examples of inorganic peroxides usable in the method described above include hydrogen peroxide and sodium metaperiodate. As suitable organic peroxides, there are mentioned *m*-chloro-perbenzoic acid, perbenzoic acid and peracetic acid.

It is preferred to use 1.1 to 1.5 moles of the peroxide per 1 mole of the starting compound of formula (2).

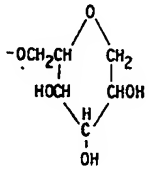
The solvent used in the reaction mixture and the time period for carrying out the reaction can be determined depending on the kind of oxidizing agent that is used. In general, the oxidizing reaction of the invention is carried out in a solvent such as a hydrated alcohol, acetic acid or a chlorinated hydrocarbon such as chloroform or methylene chloride, at a temperature of −10°C to 80°C. More specifically, when sodium metaperiodate is used, the reaction is carried out in a hydrated alcohol at 0° to 25°C; when hydrogen peroxide is used, the reaction is carried out in a hydrated alcohol at 60° to 70°C or in acetic acid at 30° to 80°C; when *m*-chloro-perbenzoic acid or perbenzoic acid is used, the reaction is carried out in a chlorinated hydrocarbon such as chloroform or methylene chloride at 0° to 25°C; and when peracetic acid is used, the reaction is carried out in acetic acid at −10° to 0°C.

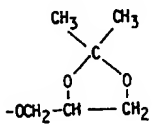
The compounds having the formula (2) which are used as starting materials for preparing the compounds of the present invention having the formula (1), can be prepared by the method of reacting mercaptans having the formula RSH (3) with acetylene-monocarboxylic acid in an aqueous solution of an alkali metal hydroxide to form compounds having the formula (4):

$$RS—CH=CH—COOM \qquad (4)$$

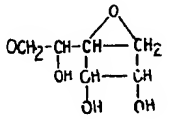
wherein R is as defined above and M is hydrogen or alkali metal. The compounds obtained by esterifying or forming amides of the compounds having the formula (4) can also be used as starting materials for preparing the compounds of the present invention.

Among the groups represented by —COY for X in the compounds of the formula (2) and used as starting materials for the compounds of the invention, hydroxyl-substituted alkoxy groups are formed by removing one hydrogen atom from one hydroxyl group of a polyfunctional alcohol, especially a saturated, aliphatic or alicyclic alcohol having from 2 to 10 carbon atoms. The cyclic groups





and



having ether bonds located in the ring, can also be used. Such alkoxy groups are formed by removing one hydrogen atom from one hydroxyl group of a polyfunctional alcohol such as ethylene glycol, propylene glycol, glycerin, erythritol, pentaerythritol, xylitol, sorbitol, mannitol, diglycerin, dipentaerythritol, xylitan, sorbitan, mannitan and polyethylene glycols.

The compounds of the present invention have the formula (1) are used for germicides or sterilizers other than for medical uses, and also as antifungal agents, and antiseptics. The compounds of the present invention prevent growth of Gram positive organisms such as Staphylococcus aureus and Bacillus subtilis which are representative micro-organisms that cause various injuries under normal living environments, and also prevent growth of the Gram negative organisms such as Escherichia coli, Proteus vulgaris and Pseudomonas including Pseudomonas aeruginosa which is well known as a representative putrefactive bacterium. The compounds of the invention also have the function of preventing growth of various molds such as Penicillium, Aspergillus and Sozopus and are further effective against yeasts belonging to the Candida genus which causes moniliasis.

The compounds of the present invention can be changed in their physiochemical properties and their antibiotic activities by introducing different groups R and X into

the formula (1) and/or by changing the number denoted by n in the formula. According to the present invention, it is thus possible to select the most suitable compound having a special structure and to add or apply it to a particular system of microorganisms. As a result, the disadvantages of the conventional antibiotic chemicals, i.e., that they cannot be applied to some systems due to their poor mutual solubility irrespective of the appreciable antibiotic activities thereof, can be conveniently overcome by the present invention.

Because of the aforementioned properties, the compounds of the present invention can be used as additives to various materials, such as various kinds of emulsion type cutting oils, detergents, soaps, shampoos, rinses, hair treatments, lotions, cosmetic goods, textile finishing agents, textile oils, paints and printing inks.

Preparation 1.

This Preparation relates to the synthesis of β-laurylmercapto-acrylic acid.

0.50 mol of sodium hydroxide was dissolved in 500 ml of water. 0.55 mol of acetylene-monocarboxylic acid was added to the solution dropwise. 0.50 mol of laurylmercaptan was added to the solution which was then agitated at room temperature for 3 hours. The reaction mixture was then neutralized with dilute hydrochloric acid and the formed product was extracted with benzene. A crystallized compound was obtained at a yield of 93% (based on the used mercaptan). The obtained crystallized compound was then subjected to infrared analysis, to nuclear magnetic resonance analysis and elementary analysis and the following results were obtained:

IR: 1660 (C=O) cm^{-1}

NMR (CCl_4, TMS): δ 7.25 (doublet, 1H, =CH—COO—),
5.85 ppm (doublet, 1H, —S—CH=).

Result of the elementary analysis:

	found	calcd.
C (%)	66.3	66.1
H (%)	10.3	10.4

From the results of the above analyses, the above crystallized material was identified as having the following structural formula:

$$n\text{—}C_{12}H_{25}S\text{—}CH\text{=}CH\text{—}COOH$$

Preparation 2.

This Preparation relates to the synthesis of β-lauryl-mercapto-acrylic acid methyl ester.

0.3 mol of β-lauryl-mercapto-acrylic acid, synthesized in Preparation 1, 30 mols of methanol and 5.0 ml of concentrated sulfuric acid were dissolved in 400 ml of benzene and heated at 80°C for 50 hours. During the heating time period, water was removed from the mixture by utilizing benzene-water azeotropy. After the completion of the reaction, the reaction mixture was concentrated under reduced pressure to obtain a crystallized compound at a yield of 76%.

The above crystallized compound was subjected to analyses and the following results were obtained.

Melting Point: 48—50°C (from hexane)

IR: 1710 (C=O) cm^{-1}

NMR (CCl_4, TMS): δ 7.55 (doublet, 1H, =CH—COO—),
5.80 ppm (doublet, 1H, —S—CH=).

Result of the elementary analysis:

	found	calcd.
C (%)	67.2	67.1
H (%)	10.4	10.6

From the results of the above analyses, the above crystallized compound was identified as having the following structural formula:

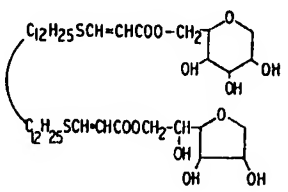
$$n\text{—}C_{12}H_{25}S\text{—}CH\text{=}CH\text{—}COOCH_3$$

Preparation 3.

Various esters of β-mercapto-acrylic acids were synthesized. The conditions and yields of the reactions are shown in Table 1, and the properties of the products are shown in Table 2.

TABLE 1

Preparation of esters of β-mercapto acrylic acids

R in β-mercapto acrylic acid	Radical of alcohol deprived of one OH group	Quantity of alcohol in mol added to 0.30 mol of mercapto acrylic acid	Catalyst	Quantity of the catalyst	Solvent	Reaction temp. (°C)	Reaction period (hr)	Product	Yield based on mercapto acrylic acid
$C_{12}H_{25}$	CH_3	3.0	H_2SO_4	5ml	benzene (400m)	80	5	$C_{12}H_{25}S-CH=CH-COOCH_3$	76
	$-CH_2CH(OH)-CH_2(OH)$	2.0	Amberlite* IR–120	10 g	toluene (400m)	110	5	$C_{12}H_{25}S-CH=CH-COOCH_2-CH(OH)-CH_2(OH)$	68
	$-CH_2-(-CH(OH)-)_4-CH_2OH$	3.0	Amberlite IR–120	10 g	toluene (400m)	110	5		48 48
	$-(CH_2CH_2O)_2CH_3$	2.0	H_2SO_4	5ml	benzene (400m)	80	5	$C_{12}H_{25}SCH=CHCOO(CH_2CH_2O)_2CH_3$	78
CH_3	CH_3	3.0	H_2SO_4	5ml	benzene (400m)	80	5	$CH_3SCH=CHCOOCH_3$	80

* 'Amberlite' is a Regd. Trade Mark.

TABLE 1 (Continued)

Preparation os esters of β-mercapto acrylic acids

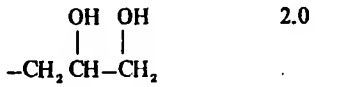
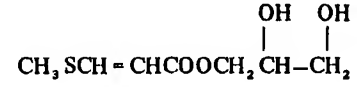
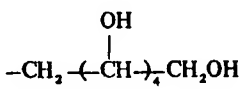
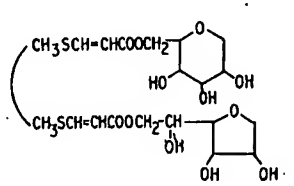
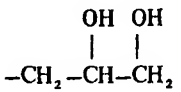
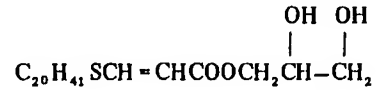
R in β-mercapto acrylic acid	Radical of alcohol deprived of one OH group	Quantity of alcohol in mol added to 0.30 mol of mercapto acrylic acid	Catalyst	Quantity of the catalyst	Solvent	Reaction temp. (°C)	Reaction period (hr)	Product	Yield based on mercapto acrylic acid
		2.0	Amberlite IR–120	10 g	toluene (400m)	110	5		76
		3.0	Amberlite IR–120	10 g	toluene (400m)	110	5		32 33
	$-(CH_2CH_2O)_2CH_3$	2.0	H_2SO_4	5ml	benzene (400m)	80	5	$CH_3SCH=CHCOO(CH_2CH_2O)_2CH_3$	68
$C_{20}H_{41}$	CH_3	3.0	H_2SO_4	5ml	benzene (400m)	80	5	$C_{20}H_{41}SCH=CHCOOCH_3$	75
		2.0	Amberlite IR–120	10 g	toluene (400m)	110	5		60

TABLE 1 (Continued)

Preparation of esters of β-mercapto acrylic acids

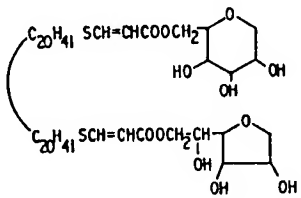
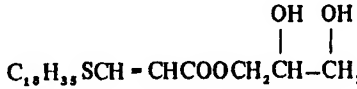
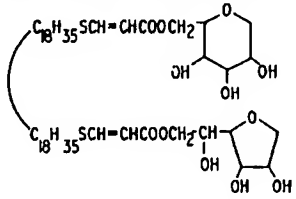
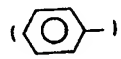
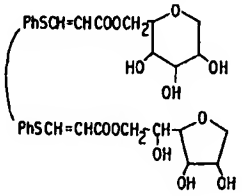
R in β-mercapto acrylic acid	Radical of alcohol deprived of one OH group	Quantity of alcohol in mol added to 0.30 mol of mercapto acrylic acid	Catalyst	Quantity of the catalyst	Solvent	Reaction temp. (°C)	Reaction period (hr)	Product	Yield based on mercapto acrylic acid
		3.0	Amberlite IR–120	10 g	toluene (400m)	110	5		40 45
	$-(CH_2CH_2O)_2CH_3$	2.0	H_2SO_4	5ml	benzene (400m)	80	5	$C_{20}H_{41}SCH=CHCOO(CH_2CH_2O)_2CH_3$	82
Oleyl ($C_{18}H_{15}$)	CH_3	3.0	H_2SO_4	5ml	benzene (400m)	80	5	$C_{18}H_{35}SCH=CHCOOCH_3$	78
		2.0	Amberlite IR–120	10 g	toluene (400m)	110	5		68
		3.0	Amberlite IR–120	10 g	toluene (400m)	110	5		38 36

TABLE 1 (Continued)

Preparation of esters of β-mercapto acrylic acds

R in β-mercapto acrylic acid	Radical of alcohol deprived of one OH group	Quantity of alcohol in mol added ro 0.30 mol of mercapto acrylic acid	Catalyst	Quantity of the catalyst	Solvent	Reaction temp. (°C)	Reaction period (hr)	Product	Yield based on mercapto acrylic acid
	$-(CH_2CH_2O)_2CH_3$	2.0	H_2SO_4	5ml	benzene (400m)	80	5	$C_{18}H_{35}SCH=CHCOO(CH_2CH_2O)_2CH_3$	72
Phenyl	CH_3	3.0	H_2SO_4	5ml	benzene (400m)	80	5	$PhSCH=CHCOOCH_3$	80
		2.0	Amberlite IR–120	10 g	toluene (400m)	110	5		78
		3.0	Amberlite IR–120	10 g	toluene (400m)	110	5		29 30
	$-(CH_2CH_2O)_2CH_3$	2.0	H_2SO_4	5ml	benzene (400m)	80	5	$PhSCH=CHCOO(CH_2CH_2O)_2CH_3$	69

8

TABLE 2

Properties of esters of β-mercapto acrylic acids

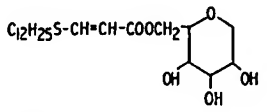
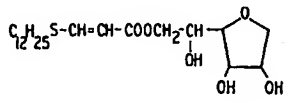
Compound	Property	IR (cm^{-1})	NMR (CCl_4, TMS, δ ppm)	Result of elementary analysis found C (%)	found H (%)	calcd C (%)	calcd H (%)
$C_{12}H_{25}S-CH=CH-COOCH_3$	mp. 48–50°C (from hexane)	1710 (C=O)	7.55 (doublet, 1H, =CH–COO–), 5.80 (doublet, 1H, –S–CH=)	67.2	10.4	67.1	10.6
$C_{12}H_{25}S-CH=CH-COOCH_2-CH(OH)-CH_2(OH)$	liquid	3300 (–OH) 1715 (C=O)	7.34 (doublet, 1H, =CH–COO–), 5.75 (doublet, 1H, –S–CH=)	62.3	10.3	62.4	10.0
	mp. 43–46°C (from hexane)	3300 (–OH) 1710 (C=O)	7.38 (doublet, 1H, =CH–COO–), 5.81 (doublet, 1H, –S–CH=)	62.4	9.3	62.7	9.5
	mp. 45–47°C (from hexane)	3300 (–OH) 1713 (C=O)	7.39 (doublet, 1H, =CH–COO–), 5.78 (doublet, 1H, –S–CH=)	62.1	9.4	62.7	9.5
$C_{12}H_{25}S-CH=CH-COO(CH_2CH_2O)_2CH_3$	liquid	3300 (–OH) 1710 (C=O)	7.40 (doublet, 1H, =CH–COO–), 5.80 (doublet, 1H, –S–CH=)	63.9	10.3	64.1	10.2
$CH_3SCH=CHCOOCH_3$	liquid	1712 (C=O)	7.40 (doublet, 1H, =CH–COO–), 5.92 (doublet, 1H, –S–CH=)	45.3	5.9	45.5	6.1

TABLE 2 (Continued)

Properties of esters of β-mercapto acrylic acids

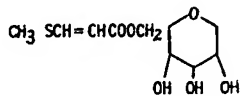
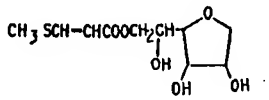
Compound	Property	IR (cm^{-1})	NMR (CCl_4, TMS, δ ppm)	Result of elementary analysis found C (%)	found H (%)	calcd C (%)	calcd H (%)
$CH_3SCH=CHCOOCH_2CH(OH)CH_2(OH)$	liquid	3300 (OH) 1715 (C=OL	7.36 (doublet, 1H, =CH–COO–), 5.70 (doublet, 1H, –S–CH=)	44.2	6.3	43.8	6.3
	liquid	3300 (OH) 1710 (C=O)	7.41 (doublet, 1H, =CH–COO–), 5.78 (doublet, 1H, –S–CH=)	45.6	6.0	45.5	6.1
	liquid	3300 (OH) 1714 (C=O)	7.38 (doublet, 1H, =CH–COO–), 5.70 (doublet, 1H, –S–CH=)	45.3	6.0	45.5	6.1
$CH_3SCH=CHCOO(CH_2CH_2O)_2CH_3$	liquid	1710 (C=O)	7.63 (doublet, 1H, =CH–COO–), 5.62 (doublet, 1H, –S–CH=)	49.3	7.1	49.1	7.3
$C_{20}H_{41}SCH=CHCOOCH_3$	mp. 40–41°C (from hexane)	1715 (C=O)	7.36 (doublet, 1H, =CH–COO–), 5.62 (doublet, 1H, –S–CH=)	72.1	11.3	72.3	11.6
$C_{20}H_{41}SCH=CHCOOCH_2CH(OH)CH_2(OH)$	mp. 43–44°C (from hexane)	3300 (OH) 1715 (C=O)	7.40 (doublet, 1H, =CH–COO–), 5.70 (doublet, 1H, –S–CH=)	68.4	10.8	68.1	11.0

TABLE 2 (Continued)

Properties of esters of β-mercapto acrylic acids

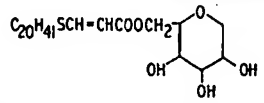
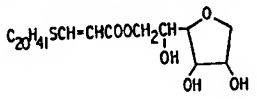
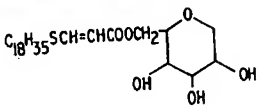
Compound	Property	IR (cm^{-1})	NMR (CCl_4, TMS, δ ppm)	Result of elementary analysis found C (%)	found H (%)	calcd C (%)	calcd H (%)
	m.p. 50–51°C (from hexane)	3300 (OH) 1710 (C=O)	7.42 (doublet, 1H, =CH–COO–), 5.36 (doublet, 1H, –S–CH=)	65.4	10.3	65.6	10.3
	mp. 53–54°C (from hexane)	3300 (OH) 1714 (C=O)	7.20 (doublet, 1H, =CH–COO–), 5.72 (doublet, 1H, –S–CH=)	65.3	10.2	65.6	10.3
$C_{20}H_{41}SCH=CHCOO(CH_2CH_2O)_2CH_3$	mp. 48–49°C (from hexane)	1710 (C=O)	7.38 (doublet, 1H, =CH–COO–), 5.62 (doublet, 1H, –S–CH=)	69.4	11.0	69.1	11.2
$C_{18}H_{35}SCH=CHCOOCH_3$	mp. 38–39°C (from hexane)	1710 (C=O)	7.39 (doublet, 1H, =CH–COO–), 5.29 (doublet, 1H, –S–CH=)	71.6	10.8	71.7	10.9
$C_{18}H_{35}SCH=CHCOOCH_2CHCH_2$ (OH OH)	mp. 40–41°C (from hexane)	3300 (OH) 1710 (C=O)	7.62 (doublet, 1H, =CH–COO–), 5.43 (doublet, 1H, –S–CH=)	67.1	10.3	67.2	10.3
	mp. 43–44°C (from hexane)	3300 (OH) 1710 (C=O)	7.37 (doublet, 1H, =CH–COO–), 5.40 (doublet, 1H, –S–CH=)	64.9	9.7	64.8	9.7

TABLE 2 (Continued)

Properties of esters of β-mercapto acrylic acids

Compound	Property	IR (cm^{-1})	NMR (CCl_4, TMS, δ ppm)	Result of elementary analysis found C (%)	found H (%)	calcd C (%)	calcd H (%)
$PhSCH=CHCOO(CH_2CH_2O)_2CH_3$	liquid	1710 (C=O)	7.36 (doublet, 1H, =CH–COO–) 5.42 (doublet, 1H, –S–CH=)	59.7	6.4	59.6	6.4

Example 1.

0.33 mol of sodium metaperiodate ($NaIO_4$) was dissolved in a mixed solvent containing 200 ml of H_2O and 200 ml of MeOH. 0.30 mol of β-lauryl-mercapto-acrylic acid was added to the solution at room temperature. After agitating the mixture at 25°C for 24 hours, the formed sodium iodate ($NaIO_3$) was filtered off. The filtrate was extracted with diethyl ether. A crystallized compound was obtained at a yield of 88%. The results of the analyses of the crystallized compound are set forth below.

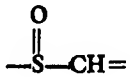
Melting Point: 62—65°C (from hexane)

IR (cm^{-1}): 3300 (OH),

1690 (),

1050 (S=O)

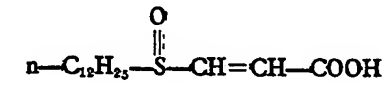
NMR (CCl_4, TMS): 7.25 (doublet, 1H, =CH—COO—),
6.60 ppm (doublet, 1H,

).

Result of the elementary analysis:

	found	calcd.
C (%)	62.4	62.7
H (%)	7.3	7.5

From the results of the above analyses, the above crystallized compound was identified as having the following structural formula:



Example 2.

0.30 mol of β-lauryl-mercapto-acrylic acid was dissolved in 300 ml of EtOH. 0.50 mol of hydrogen peroxide in the form of a 30% aqueous solution was added and the mixture was heated at 70°C for 48 hours under agitation. The reaction mixture was extracted with diethyl ether to obtain a crystallized compound (yield: 68%). The results of the analytical tests confirmed that the crystallized compound obtained in this Example was the same as that of Example 1.

Example 3.

0.30 mol of β-lauryl-mercapto-acrylic acid was dissolved in 200 ml of chloroform. 0.33 mol of *m*-chloroperbenzoic acid in 100 ml of chloroform was added to the solution at 25°C. After agitating the mixture for 1 hour, the reaction product was extracted with diethyl ether. A crystallized compound was obtained (yield: 90%). The results of the analytical tests confirmed that the crystallized compound obtained in this Example was the same as that of Example 1.

Example 4.

0.30 mol of β-lauryl-mercapto-acrylic acid was dissolved in 200 ml of AcOH. 0.30 mol of peracetic acid was added to the solution at −10°C, and the mixture was agitated for 1 hour. Water was poured into the reaction mixture and the reaction product was extracted with diethyl ether. A crystallized compound was obtained (yield: 70%). The results of the analytical tests confirmed that the crystallized compound obtained in this Example was the same as that of Example 1.

Example 5.

Various β-sulfinyl-acrylic acids, esters thereof and amides thereof were synthesized. The reaction conditions and the yields are set forth in the following Table 7, and the properties of the obtained products are set forth in Table 8.

TABLE 7

Preparation of β-sulfinyl acrylic acids and esters and amides thereof

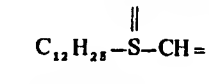
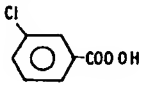
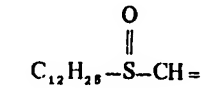
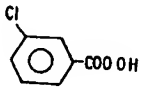
Compound (2) R:	Compound (2) X:	Oxidizing agent	Quantity of oxidizing agent in moles added to 0.30 mols of the Compound (2)	Solvent	Reaction period (hr.)	Reation temp. (°C)	Yield (based on compound (2), (%)	Product
$-C_{12}H_{25}$		$NaIO_4$	0.33	MeOH(200ml)–H_2O(200ml)	24	25	88	
		H_2O_2	0.50	EtOH(300ml)	48	80	68	
			0.33	$CHCl_3$(300ml)	1	25	90	
		CH_3COOOH	0.30	AcOH(200ml)	1	–10	70	
		$NaIO_4$	0.33	MeOH(200ml)–H_2O(200ml)	24	25	79	
		H_2O_2	0.50	EtOH(300ml)	48	80	52	
			0.33	$CHCl_3$(300ml)	1	25	92	
		CH_3COOOH	0.30	AcOH(200ml)	1	–10	64	

TABLE 7 (Continued)

Preparation of β-sulfinyl acrylic acids and esters and amides thereof

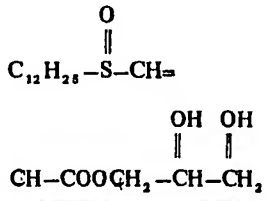
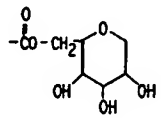
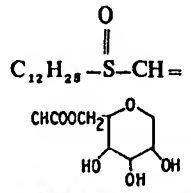
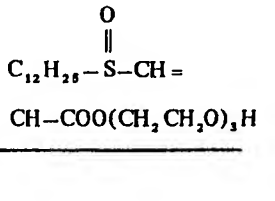
R:	Compound (2) X:	Oxidizing agent	Quantity of oxidizing agent in moles added to 0.30 mols of the Compound (2)	Solvent	Reaction period (hr.)	Reaction temp. (°C)	Yield (based on compound (2), (%)	Product
		$NaIO_4$	0.33	MeOH(200ml)–H_2O(200ml)	24	25	82	
		H_2O_2	0.50	EtOH(300ml)	48	80	76	
		$NaIO_4$	0.33	MeOH(200ml)–H_2O(200ml)	24	25	74	
		H_2O_2	0.50	EtOH(300ml)	48	80	68	
		$NaIO_4$	0.33	MeOH(200ml)–H_2O(200ml)	24	25	76	
		H_2O_2	0.50	EtOH(300ml)	48	80	59	

TABLE 7 (Continued)

Preparation of β-sulfinyl acrylic acids and esters and amides thereof

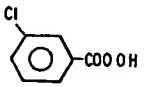
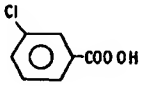
Compound (2) R:	Compound (2) X:	Oxidizing agent	Quantity of oxidizing agent in moles added ro 0.30 mols of the Compound (2)	Solvent	Reaction period (hr.)	Reaction temp. (°C)	Yield (based on compound (2), (%)	Product
	$-CO(CH_2CH_2O)_2Me$	$NaIO_4$	0.33	$MeOH(200ml)-H_2O(200ml)$	24	25	83	
			0.33	$CHCl_3(300ml)$	1	25	94	$C_{12}H_{25}S-CH=CH-COO(CH_2CH_2O)_2Me$
	$-CN(CH_2CH_2OH)_2$	$NaIO_4$	0.33	$MeOH(200ml)-H_2O(200ml)$	24	25	70	$C_{12}H_{25}S-CH=$
			0.33	$CHCl_3(300ml)$	1	25	82	$CH-CON(CH_2CH_2OH)_2$
	$-CNHCH_2CH_2SO_3Na$	H_2O_2	0.50	$MeOH(200ml)-H_2O(200ml)$	48	80	76	$C_{12}H_{25}S-CH=CH-CNHCH_2CH_2SO_3Na$

TABLE 7 (Continued)

Preparation of β-sulfinyl acrylic acids and esters and amides thereof

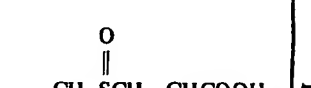
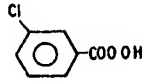
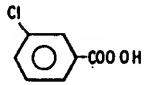
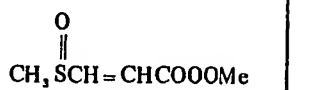
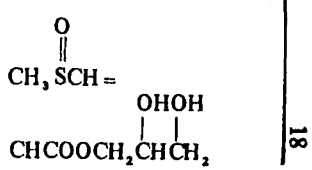
Compound (2) R:	Compound (2) X:	Oxidizing agent	Quantity of oxidizing agent in moles added to 0.30 mols of the Compound (2)	Solvent	Reaction period (hr.)	Reaction temp. (°C)	Yield (based on compound (2), (%)	Product
CH_3		$NaIO_4$	0.33	MeOH(200ml)–H_2O(200ml)	24	25	85	
			0.50	EtOH(300ml)	48	80	70	
			0.33	$CHCl_3$(300ml)	1	25	63	
		CH_3COOOH	0.30	AcOH(200ml)	1	−10	72	
		$NaIO_4$	0.33	MeOH(200ml)–H_2O(200ml)	24	25	76	
		H_2O_2	0.50	EtOH(300ml)	48	80	48	
			0.33	$CHCl_3$(300ml)	1	25	68	
		CH_3COOOH	0.30	AcOH(200ml)	1	−10	70	
		$NaIO_4$	0.33	MeOH(200ml)–H_2O(200ml)	24	25	75	
		H_2O_2	0.50	EtOH(300ml)	48	80	63	

TABLE 7 (Continued)

Preparation of β-sulfinyl acrylic acids and esters and amides thereof

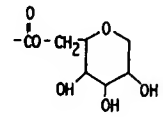
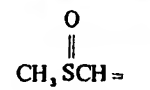
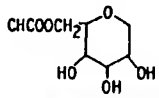
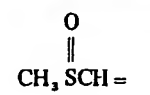
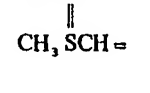
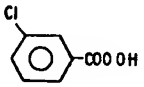
Compound (2) R:	Compound (2) X:	Oxidizing agent	Quantity of oxidizing agent in moles added to 0.30 mols of the Compound (2)	Solvent	Reaction period (hr.)	Reaction temp. (°C)	Yield (based on compound (2), (%)	Product
		$NaIO_4$	0.33	MeOH(200ml)–H_2O(200ml)	24	25	72	
		H_2O_2	0.50	EtOH(300ml)	48	80	63	
	$-CO(CH_2CH_2O)_3H$	$NaIO_4$	0.33	MeOH(200ml)–H_2O(200ml)	24	25	73	
		H_2O_2	0.50	EtOH(300ml)	48	80	56	$CHCOO(CH_2CH_2O)_3H$
	$-CO(CH_2CH_2O)_2CH_3$	$NaIO_4$	0.33	MeOH(200ml)–H_2O(200ml)	24	25	80	
			0.33	$CHCl_3$(300ml)	1	25	83	$CHCOO(CH_2CH_2O)_2CH_3$

TABLE 7 (Continued)

Preparation of β-sulfinyl acrylic acids and esters and amides thereof

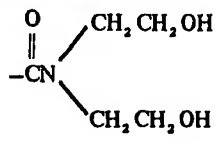
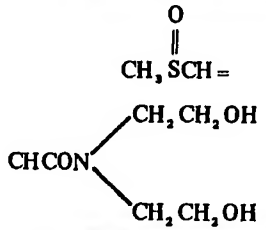
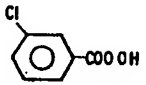
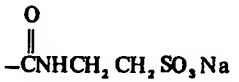
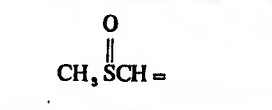
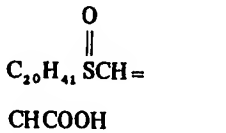
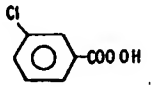
	$NaIO_4$	0.33	MeOH(200ml)–H_2O(200ml)	24	25	68	
		0.33	$CHCl_3$(300ml)	1	25	75	
	H_2O_2	0.50	MeOH(200ml)–H_2O(200ml)	48	80	73	
$C_{20}H_{41}$ 	$NaIO_4$	0.33	MeOH(200ml)–H_2O(200ml)	24	25	85	
	H_2O_2	0.50	EtOH(300ml)	48	80	70	
		0.33	$CHCl_3$(300ml)	1	25	63	
	CH_3COOOH	0.30	AcOH(200ml)	1	−10	72	

TABLE 7 (Continued)

Preparation of β-sulfinyl acrylic acids and esters and amides thereof

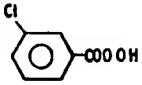
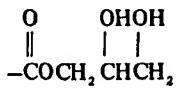
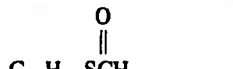
Compound (2) R:	Compound (2) X:	Oxidizing agent	Quantity of oxidizing agent in moles added to 0.30 mols of the Compound (2)	Solvent	Reaction period (hr.)	Reaction temp. (°C)	Yield (based on compound (2), (%)	Product
		$NaIO_4$	0.33	MeOH(200ml)–H_2O(200ml)	24	25	76	
		H_2O_2	0.50	EtOH(300ml)	48	80	49	
			0.33	$CHCl_3$(300ml)	1	25	70	$CHCOOCH_3$
		CH_3COOOH	0.30	AcOH(200ml)	1	−10	68	
		$NaIO_4$	0.33	MeOH(200ml)–H_2O(200ml)	24	25	75	
		H_2O_2	0.50	EtOH(300ml)	48	80	65	

TABLE 7 (Continued)

Preparation of β-sulfinyl acrylic acids and esters and amides thereof

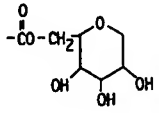
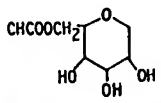
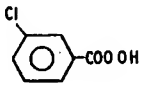
Compound (2) R:	Compound (2) X:	Oxidizing agent	Quantity of oxidizing agent in moles added to 0.30 mols of the Compound (2)	Solvent	Reaction period (hr.)	Reaction temp. (°C)	Yield (based on compound (2), (%)	Product
		$NaIO_4$	0.33	MeOH(200ml)–H_2O(200ml)	24	25	68	
		H_2O_2	0.50	EtOH(300 ml)	48	80	60	
	$-CO(CH_2CH_2O)_3H$	$NaIO_4$	0.33	MeOH(200ml)–H_2O (200ml)	24	25	72	
		H_2O_2	0.50	EtOH(300ml)	48	80	55	$CHCOO(CH_2CH_2O)_3H$
	$-CO(CH_2CH_2O)_2CH_3$	$NaIO_4$	0.33	MeOH(200ml)–H_2O(200m)	24	25	80	
			0.33	$CHCl_3$(300ml)	1	25	68	$CHCOO(CH_2CH_2O)_2CH_3$

TABLE 7 (Continued)

Preparation of β-sulfinyl acrylic acids and esters and amides thereof

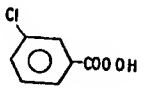
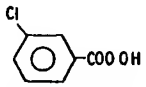
R:	Compound (2) X:	Oxidizing agent	Quantity of oxidizing agent in moles added to 0.30 mols of the Compound (2)	Solvent	Reaction period (hr.)	Reaction temp. (°C)	Yield (based on compound (2), (%)	Product
	$-CN(CH_2CH_2OH)_2$	$NaIO_4$	0.33	MeOH(200ml)–H_2O(200ml)	24	25	70	$C_{20}H_{41}SCH=C(CN)(CH_2CH_2OH)_2$
			0.33	$CHCl_3$(300ml)	1	25	82	$CHCON(CH_2CH_2OH)_2$
	$-CNHCH_2CH_2SO_3Na$	H_2O_2	0.50	MeOH(200ml)–H_2O(200ml)	48	80	73	$C_{20}H_{41}SCH=CHCONHCH_2CH_2SO_3Na$
Oleyl ($C_{18}H_{35}$)		$NaIO_4$	0.33	MeOH(200ml)–H_2O(200ml)	24	25	80	$C_{18}H_{35}SCH=$
	$-C-OH$	H_2O_2	0.50	EtOH(300ml)	48	80	65	CHCOOH
			0.33	$CHCl_3$(300ml)	1	25	63	
		CH_3COOOH	0.30	AcOH(200ml)	1	−10	70	

TABLE 7 (Continued)

Preparation of β-sulfinyl acrylic acids and esters and amides thereof

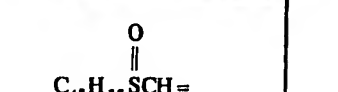
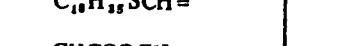
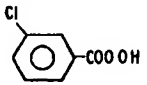
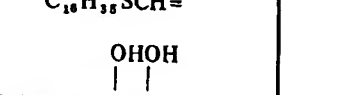
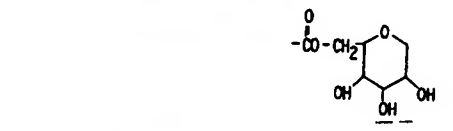
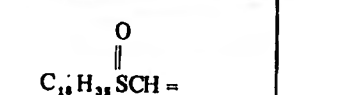
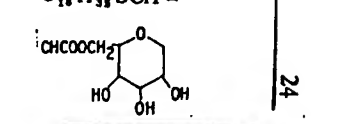
Compound (2) R:	Compound (2) X:	Oxidizing agent	Quantity of oxidizing agent in moles added to 0.30 mols of the Compound (2)	Solvent	Reaction period (hr.)	Reaction temp. (°C)	Yield (based on compound (2), (%)	Product
		$NaIO_4$	0.33	MeOH(200ml)–H_2O(200ml)	24	25	83	
		H_2O_2	0.50	EtOH(300ml)	48	80	62	
			0.33	$CHCl_3$(300ml)	1	25	53	
		CH_3COOOH	0.30	AcOH(200ml)	1	−10	63	
		$NaIO_4$	0.33	MeOH(200ml)–H_2O(200ml)	24	25	73	
		H_2O_2	0.50	EtOH(300ml)	48	80	48	
		$NaIO_4$	0.33	MeOH(200ml)–H_2O(200ml)	24	25	72	
		H_2O_2	0.50	EtOH(300ml)	48	80	63	

TABLE 7 (Continued)

Preparation of β-sulfinyl acrylic acids and esters and amides thereof

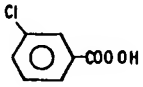
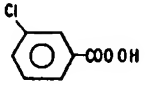
R:	Compound (2) X:	Oxidizing agent	Quantity of oxidizing agent in moles added to 0.30 mols of the Compound (2)	Solvent	Reaction period (hr.)	Reaction temp. (°C)	Yield (based on compound (2), (%)	Product
	$-CO(CH_2CH_2O)_3H$	$NaIO_4$	0.33	MeOH(200ml)–H_2O(200ml)	24	25	82	$C_{18}H_{35}SCH=CHCOO(CH_2CH_2O)_3H$
		H_2O_2	0.50	EtOH(300ml)	48	80	70	
	$-CO(CH_2CH_2O)_2Me$	$NaIO_4$	0.33	MeOH(200ml) H_2O(200ml)	24	25	63	$C_{18}H_{35}SCH=CHCOO(CH_2CH_2O)_2Me$
			0.33	$CHCl_3$(300ml)	1	25	70	
	$-CN(CH_2CH_2OH)_2$	$NaIO_4$	0.33	MeOH(200ml)–H_2O(200ml)	24	25	72	$C_{18}H_{35}SCH=CHCON(CH_2CH_2OH)_2$
			0.33	$CHCl_3$(300ml)	1	25	65	

TABLE 7 (Continued)

Preparation of β-sulfinyl acrylic acids and esters and amides thereof

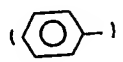
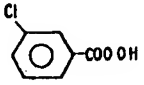
Compound (2) R:	Compound (2) X:	Oxidizing agent	Quantity of oxidizing agent in moles added to 0.30 mols of the Compound (2)	Solvent	Reaction period (hr.)	Reaction temp. (°C)	Yield (based on compound (2), (%)	Product
	O ‖ –CNHCH$_2$CH$_2$SO$_3$Na	H_2O_2	0.50	MeOH(200ml)–H_2O(200ml)	48	80	43	O ‖ $C_{18}H_{35}SCH$= CHCONHCH$_2$CH$_2$SO$_3$Na
Ph		NaIO	0.33	MeOH(200ml)–H_2O(200ml)	24	25	63	O ‖ PhSCH=
	O ‖ –COH	H_2O_2	0.50	EtOH(300ml)	48	80	70	CHCOOH
			0.33	$CHCl_3$(300ml)	1	25	82	
		CH_3COOOH	0.30	AcOH(200ml)	1	–10	65	
	O ‖ –COMe	$NaIO_4$	0.33	MeOH(200ml)–H_2O(200ml)	24	25	82	O ‖ PhSCH=
		H_2O_2	0.50	EtOH(300ml)	48	80	62	$CHCOOCH_3$

TABLE 7 (Continued)

Preparation of β-acrylic acids and esters and amides thereof

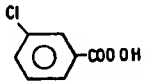
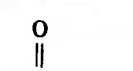
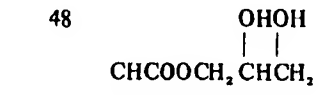
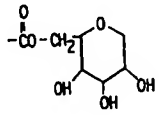
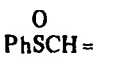
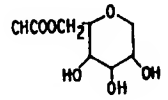
Compound (2) R:	Compound (2) X:	Oxidizing agent	Quantity of oxidizing agent in moles added to 0.30 moles of the Compound (2)	Solvent	Reaction period (hr.)	Reaction temp. (°C)	Yield (based on compound (2), (%)	Product
			0.33	$CHCl_3$(300ml)	1	25	85	
		CH_3COOOH	0.30	AcOH(200ml)	1	−10	70	
		$NaIO_4$	0.33	MeOH(200ml)−H_2O(200ml)	24	25	76	
		H_2O_2	0.50	EtOH(300ml)	48	80	48	
		$NaIO_4$	0.33	MeOH(200ml)−H_2O(200ml)	24	25	68	
		H_2O_2	0.50	EtOH(300ml)	48	80	52	

TABLE 7 (Continued)

Preparation of β-acrylic acids and esters and amides thereof

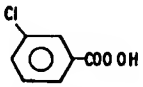
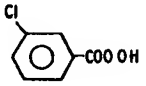
Compound (2) R:	Compound (2) X:	Oxidizing agent	Quantity of oxidizing agent in moles added to 0.30 moles of the Compound (2)	Solvent	Reaction period (hr.)	Reaction temp. (°C)	Yield (based on compound (2), (%)	Product
	$-CO(CH_2CH_2O)_3H$	$NaIO_4$	0.33	MeOH(200ml)–H_2O(200ml)	24	25	73	PhSCH=
		H_2O_2	0.50	EtOH(300ml)	48	80	61	$CHCOO(CH_2CH_2O)_3H$
	$-CO(CH_2CH_2O)_2Me$	$NaIO_4$	0.33	MeOH(200ml)–H_2O(200ml)	24	25	70	PhSCH=
			0.33	$CHCl_3$(300ml)	1	25	64	$CHCOO(CH_2CH_2O)_2Me$
	$-CN(CH_2CH_2OH)_2$	$NaIO_4$	0.33	MeOH(200ml)–H_2O(200ml)	24	25	73	PhSCH=
			0.33	$CHCl_3$(300ml)	1	25	75	$CHCON(CH_2CH_2OH)_2$

TABLE 7 (Continued)

Preparation of β-acrylic acids and esters and amides thereof

Compound (2) R:	Compound (2) X:	Oxidizing Agent	Quantity of oxidizing agent in moles added to 0.30 mols of the Compound (2)	Solvent	Reaction period (hr.)	Reaction temp. (°C)	Yield (based on compound (2), (%)	Product
	$-\overset{\overset{O}{\|}}{C}NHCH_2CH_2SO_3Na$	H_2O_2	0.50	MeOH(200ml)–H_2O(200ml)	48	80	70	$PhSCH=\overset{\overset{O}{\|}}{C}HCONHCH_2CH_2SO_3Na$

TABLE 8 : PROPERTIES OF β-SULFINYL-ACRYLIC ACIDS AND ESTERS AND AMIDES THEREOF

COMPOUND	PROPERTY	IR (cm^{-1})	NMR (CCl_4, TMS, ppm)	Result of elementary analysis found C (%)	found H (%)	calcd. C (%)	calcd. N (%)
$C_{12}H_{25}-\overset{O}{\overset{\|}{S}}-CH=CH-COOH$	m.p. 62 – 65°C (from hexane)	3300 (OH) 1690 (–CO–) 1050 (–S–)	7.25 (doublet, 1H, =CH–COO 6.60 (doublet, 1H, –S–CH=)	62.4	7.3	62.5	4.5
$C_{12}H_{25}-\overset{O}{\overset{\|}{S}}-CH=CH-COOCH_3$	m.p. 55 – 53°C (from hexane)	1710 (–C–O–) 1045 (–S–)	7.20 (doublet, 1H, =CH–COO–) 6.52 (doublet, 1H, –S–CH=)	62.3	10.1	62.0	10.4
$C_{12}H_{25}-\overset{O}{\overset{\|}{S}}-CH=CH-\overset{O}{\overset{\|}{C}}-CH_2-\underset{OH}{\underset{\|}{CH}}-\underset{OH}{\underset{\|}{CH_2}}$	Liquid	3300 (OH) 1715 (–C–O–) 1050 (–S–)	7.58 (doublet, 1H, =CH–COO–) 5.60 (doublet, 1H, –S–CH-)	62.3	9.5	62.4	9.9

TABLE 8 (cont.) PROPERTIES OF β-SULFINYL-ACRYLIC ACIDS AND ESTERS AND AMIDES THEREOF

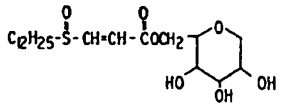
COMPOUND	PROPERTY	IR (cm^{-1})	NMR (CCl_4, TMS, ppm)	Result of elementary analysis found C (%)	found H (%)	calcd. C (%)	calcd. H (%)
	m.p. 33 – 35°C (from isopropyl alcohol)	3300 (OH) 1713 (–CO–) 1035 (–S–)	7.60 (doublet, 1H, =CH–COO) 5.30 (doublet, 1H, –S–CH=)	57.6	8.3	58.0	8.8
$C_{12}H_{25}$–S–CH=CH–CO$(CH_2CH_2O)_3$H	Liquid	3300 (OH) 1715 (–CO–) 1050 (–S–)	7.33 (doublet, 1H, =CH=COO–) 6.65 (doublet, 1H, –S–CH=)	59.8	9.3	60.0	9.6
$C_{12}H_{25}$–S–CH=CH–CN$(CH_2CH_2OH)_2$	Liquid	3300 (OH) 1630 (–CN–) 1050 (–S–)	6.95 (doublet, 1H, =CH–COO–) 5.36 (doublet, 1H, –S–CH=)	59.5	9.6	60.8	10.0
$C_{12}H_{25}$–S–CH=CH–CNH$CH_2CH_2SO_3$Na	m.p. 120 – 123°C (from isopropyl alcohol)	1625 (–CN–) 1035 (–S–) 1225, 1060 (–SO_3Na)	6.80 (doublet, 1H, =CH–COO–) 5.90 (doublet, 1H, –S–CH= in CD_3OD	50.3	7.6	50.6	8.0

TABLE 8 : PROPERTIES OF β-SULFINYL-ACRYLIC ACIDS AND ESTERS AND AMIDES THEREOF (cont.)

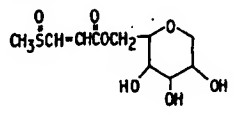
COMPOUND	PROPERTY	IR (cm^{-1})	NMR (CCl_4, TMS, ppm)	Result of elementary analysis found C (%)	found H (%)	calcd. C (%)	calcd. H (%)
	Liquid	3300 (OH) 1690 (–CO–) 1050 (–S–)	7.25 (doublet, 1H, =CH=COO–) 5.50 (doublet, 1H, –S–CH=)	35.7	4.4	35.8	4.5
	Liquid	1713 (–CO–) 1043 (–S–)	7.30 (doublet, 1H, =CH–COO–) 6.10 (doublet, 1H, –S–CH=)	40.7	5.4	40.5	5.4
	Liquid	3300 (OH) 1710 (–C–O–) 1040 (–S–)	7.25 (doublet, 1H, =CH=COO–) 5.92 (doublet, 1H, –S–CH=)	40.7	5.7	40.4	5.8
	Liquid	3300 (OH) 1715 (–C–O–) 1030 (–S–)	7.30 (doublet, 1H, =CH–COO–) 6.10 (doublet, 1H, –S–CH=)	42.6	5.8	42.9	5.8

TABLE 8: PROPERTIES OF β-SULFINYL-ACRYLIC ACIDS AND ESTERS AND AMIDES THEREOF (cont.)

COMPOUND	PROPERTY	IR (cm^{-1})	NMR (CCl_4, TMS, ppm)	Result of elementary analysis found C (%)	found H (%)	calcd. C (%)	calcd. H (%)
$CH_3SCH{=}CHCO(CH_2CH_2O)_3H$	Liquid	3300 (OH) 1710 (–CO–) 1040 (–S–)	7.11 (doublet, 1H, =CH–COO–) 5.93 (doublet, 1H, –S–CH=)	45.3	6.7	45.1	6.8
$CH_3SCH{=}CHCN(CH_2CH_2OH)_2$	Liquid	3300 (OH) 1650 (–CN–) 1045 (–S–)	7.21 (doublet, 1H, =CH=COO–) 5.93 (doublet, 1H, –S–CH=)	43.8	6.7	43.4	6.8
$CH_3SCH{=}CH{-}CNHCH_2CH_2SO_3Na$	m.p. 110–111°C (from isopropyl alcohol)	1625 (–CN–) 1040 (–S–) 1225, 1050 (–SO_3Na)	7.12 (doublet, 1H, =CH=COO–) 6.20 (doublet, 1H, –S–CH=) in CD_3OD	28.6	3.8	28.9	4.0
$C_{20}H_{41}SCH{=}CHCOOH$	m.p. 80 – 81°C (from hexane)	3300 (OH) 1685 (–CO–) 1050 (–S–)	7.25 (doublet, 1H, =CH–COO–) 5.55 (doublet, 1H, –S–CH=)	69.1	11.1	69.0	11.1

TABLE 8: PROPERTIES OF β-SULFINYL-ACRYLIC ACIDS AND ESTERS AND AMIDES THEREOF (cont.)

COMPOUND	PROPERTY	IR (cm^{-1})	NMR (CCl_4, TMS, ppm)	Result of elementary analysis found C (%)	found H (%)	calcd. C (%)	calcd. H (%)
$C_{20}H_{41}SCH{=}CHN(CH_2CH_2OH)_2$	m.p. 40–41°C (from hexane)	3300 (OH) 1645 (–CN) 1040 (–S–)	7.32 (doublet, 1H, =CH–COO–) 6.11 (doublet, 1H, –S–CH=)	66.3	10.8	66.5	10.9
$C_{20}H_{41}SCH{=}CHCNHCH_2CH_2SO_3Na$	m.p. 130–131°C (from isopropyl alcohol)	1620 (–CN–) 1035 (–S–) 1225, 1050 ($–SO_3Na$)	7.03 (doublet, 1H, =CH=COO–) 5.92 (doublet, 1H, –S–CH=) in CD_3OD	58.4	9.3	58.3	9.3
$C_{18}H_{35}SCH{=}CHCOOH$	m.p. 70–71°C (from hexane)	3300 (OH) 1680 (–CO–) 1040 (–S–)	7.30 (doublet, 1H, =CH–COO–) 5.56 (doublet, 1H, –S–CH=)	68.2	10.3	68.1	10.3
$C_{18}H_{35}SCH{=}CHCOOCH_3$	m.p. 73–74°C (from hexane)	1710 (–C–O–) 1043 (–S–)	7.12 (doublet, 1H, =CH–COO–) 6.24 (doublet, 1H, –S–CH=)	68.9	10.5	68.7	10.5

TABLE 8: PROPERTIES OF β-SULFINYL-ACRYLIC ACIDS AND ESTERS AND AMIDES THEREOF (cont.)

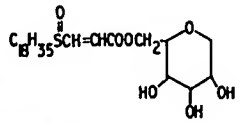
COMPOUND	PROPERTY	IR (cm^{-1})	NMR (CCl_4, TMS, ppm)	Result of elementary analysis found C (%)	found H (%)	calcd. C (%)	calcd. H (%)
$C_{18}H_{35}SCH{=}CHCOOCH_2CHCH_2$ (O; OHOH)	m.p. 58–59°C (from hexane)	3300 (OH) 1710 (–COO–) 1040 (–S–)	7.11 (doublet, 1H, =CH–COO–) 5.75 (doublet, 1H, –S–CH=)	64.9	10.2	64.8	10.0
	m.p. 55–56°C (from hexane)	3300 (OH) 1715 (–COO–) 1020 (–S–)	7.30 (doublet, 1H, =CH–COO–) 6.11 (doublet, 1H, –S–CH=)	67.8	9.6	67.7	9.7
$C_{18}H_{35}SCH{=}CHCOO(CH_2CH_2O)_3H$	m.p. 68–69°C (from hexane)	3300 (OH) 1710(–COO–) 1040 (–S–)	7.25 (doublet, 1H, =CH–COO–) 5.98 (doublet, 1H, –S–CH=)	64.7	10.1	64.5	10.0
$C_{18}H_{35}SCH{=}CHCN(CH_2CH_2OH)_2$	m.p. 38–39°C (from hexane)	3300 (OH) 1640 (–CN–) 1030 (–S–)	7.21 (doublet, 1H, =CH=COO–) 6.03 (doublet, 1H, –S–CH=)	70.8	4.2	70.3	4.0
$C_{18}H_{35}SCH{=}CHCNHCH_2CH_2SO_3Na$	m.p. 121–122°C (isopropyl alcohol)	1625 (–CON–) 1038 (–S–) 1225, 1040 ($-SO_3Na$)	7.22 (doublet, 1H, =CH=COO–) 5.93 (doublet, 1H, –S–CH=) in CD_3OD	56.6	8.3	56.9	8.6

TABLE 8: PROPERTIES OF β-SULFINYL-ACRYLIC ACIDS AND ESTERS AND AMIDES THEREOF (cont.)

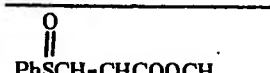
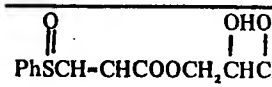
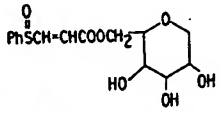
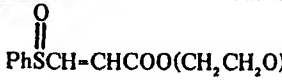
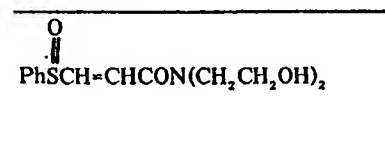
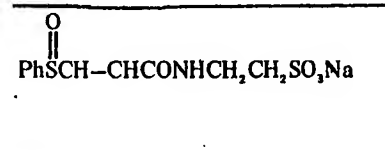
COMPOUND	PROPERTY	IR (cm^{-1})	NMR (CCl_4, TMS, ppm)	Result of elementary analysis found C (%)	found H (%)	calcd. C (%)	calcd. H (%)
	m.p. 88–89°C (from hexane)	3300 (OH) 1690 (COO) 1035 (–S–)	7.30 (doublet, 1H, =CH–COO–) 5.53 (doublet, 1H, –S–CH=)	55.3	4.2	55.1	4.1
	m.p. 80–81°C (from hexane)	1710 (COO–) 1040 (–S–)	7.22 (doublet, 1H, =CH–COO–) 6.34 (doublet, 1H, –S–CH=)	57.3	4.6	57.1	4.8
	liquid	3300 (OH) 1715 (–COO–) 1035 (–S–)	7.10 (doublet, 1H, =CH–COO–) 5.65 (doublet, 1H, –S–CH=)	53.4	5.2	53.3	5.2
	liquid	3300 (OH) 1710 (–COO–) 1030 (–S–)	7.25 (doublet, 1H, =CH–COO–) 6.01 (doublet, 1H, –S–CH=)	59.8	5.30	52.6	5.30
	liquid	3300 (OH) 1710 (–COO–) 1038 (–S–)	7.30 (doublet, 1H, =CH=COO–) 5.88 (doublet, 1H, –S–CH=)	54.6	6.1	54.9	6.1

TABLE 8: PROPERTIES OF β-SULFINYL-ACRYLIC ACIDS AND ESTERS AND AMIDES THEREOF (cont.)

COMPOUND	PROPERTY	IR (cm^{-1})	NMR (CCl_4, TMS, ppm)	Result of elementary analysis found C (%)	found H (%)	calcd. C (%)	calcd. H (%)
	Liquid	3300 (OH) 1635 (–CN–) 1038 (–S–)	7.20 (doublet, 1H, =CH=COO–) 6.13 (doublet, 1H, –S–CH=)	55.0	6.2	55.1	6.1
		1630 (–CON–) 1038 (–S–) 1225, 1040 ($-SO_3Na$)	7.24 (doublet, 1H, =CH–COO–) 5.84 (doublet, 1H, –S–CH=) in CD_3OD	42.3	3.8	42.4	3.9

Example 6.

This Example shows the antibiotic activities and the growth preventing effects of the compounds against gram positive and gram negative micro-organisms.

In accordance with the test method using agar culture media mixed with the compounds, the concentrations of the compounds of the present invention necessary for preventing growth of various organisms were determined.

1 ml of a solution of each of the compounds having predetermined concentration as set forth in the following Tables was put on a Petri dish, and 19 ml of Sabouraud's agar culture medium preliminarily heated to a molten state was then added to and uniformly mixed with the above solution, and the mixture was allowed to cool and solidify. One platinum loop of a suspension containing one million cells of an organism per 1 ml was coated on the surface of the culture medium, and was cultivated for 72 hours in a thermostatic chamber maintained at 30°C. The state of growth of the organism on each culture medium after this cultivation was observed, and the minimum concentration of the respective compounds for preventing growth of the organism on the culture medium was determined.

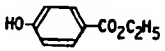
The reference symbols appearing in the following Tables have the meanings set forth hereinbelow:

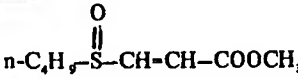
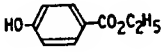
+: Growth was observed; a growth preventing effect was not exhibited.

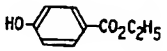
±: Growth was observed to some extent; an appreciable growth preventing effect was observed.

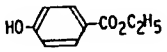
−: Growth was not observed; a perfect growth preventing effect was exhibited.

Compounds of the Invention	Organism Species	Staphylococcus aureus			Bacillus subtilis		
	Concentration of compounds (PPM)	1000	500	100	1000	500	100
		−	−	−	−	−	±
$n\text{-}C_7H_{15}CON(CH_2CH_2OH)_2$		−	+	+	−	+	+
Reference Compound							
		−	+	+	−	+	+
Reference Compound							
		−	−	+	−	±	+
Reference Compound							

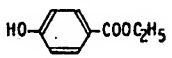
Compounds of the Invention	Organism Species	Staphylococcus aureus			Bacillus subtilis		
	Concentration of compounds (PPM)	500	100	50	500	100	50
$n\text{-}C_{12}H_{25}\text{-}\overset{\overset{O}{\|}}{S}\text{-}CH{=}CH\text{-}COOH$		–	–	–	–	–	–
$n\text{-}C_{14}H_{29}\text{-}\overset{\overset{O}{\|}}{S}\text{-}CH{=}CH\text{-}COOCH_3$		–	–	–	–	–	–
		+	+	+	+	+	+
Reference Compound							
		–	+	+	±	+	+
Reference Compound							

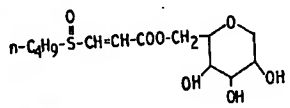
Compounds of the Invention	Organism Species / Concentration of compounds (PPM)	Escherichia coli 500	Escherichia coli 100	Escherichia coli 50	Proteus vulgaris 500	Proteus vulgaris 100	Proteus vulgaris 50	Pseudomonsa aeruginosa 500	Pseudomonsa aeruginosa 100	Pseudomonsa aeruginosa 50
		–	–	–	–	–	–	–	+	+
		+	+	+	+	+	+	+	+	+
Reference Compound										
		±	+	+	–	+	+	+	+	+
Reference Compound										

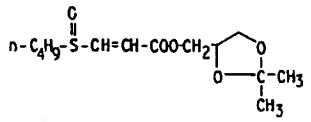
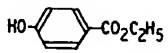
Compounds of the Invention	Organism Species	Escherichia coli			Proteus vulgaris			Pseudomonas aeruginosa		
	Concentration of compounds (PPM)	1000	500	100	1000	500	100	1000	500	100
		+	+	+	+	+	+	+	+	+
Reference Compound										
		−	±	+	−	−	+	±	+	+
Reference Compound										

Compounds of the Invention	Organism Species	Staphylococcus aureus			Bacillus subtilis			Escherichia coli			Proteus vulgaris			Pseudomonas aeruginosa		
	Concentration of compounds (PPM)	1000	500	100	1000	500	100	1000	500	100	1000	500	100	1000	500	100
		−	+	+	−	+	+	±	+	+	±	+	+	+	+	+
Reference Compound																
		−	−	+	−	±	+	−	±	+	−	−	+	±	+	+
Reference Compound																

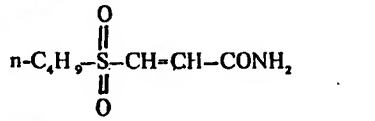
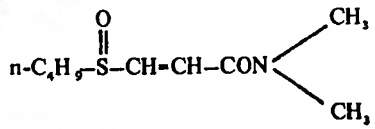
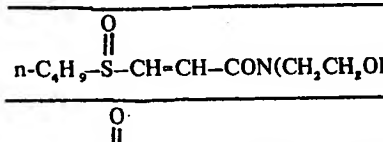
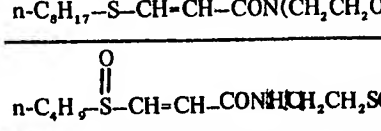
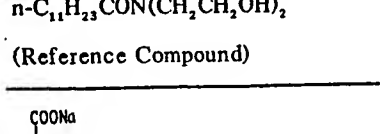
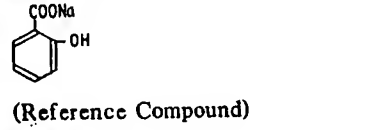
Compounds of the Invention	Organism Species	Staphylococcus aureus			Bacillus subtilis			Escherichia coli			Proteus vulgaris			Pseudomonas aeruginosa		
	Concentration of compounds (PPM)	500	100	50	500	100	50	500	100	50	500	100	50	500	100	50
$CH_3-\overset{\overset{O}{\|}}{S}-CH=CH-COOCH_3$		+	+	+	+	+	+	+	+	+	+	+	+	−	−	+
$n\text{-}C_2H_5-\overset{\overset{O}{\|}}{S}-CH=CH-COOCH_3$		±	+	+	±	+	+	±	+	+	±	+	+	−	±	+
$n\text{-}C_3H_7-\overset{\overset{O}{\|}}{S}-CH=CH-COOCH_3$		−	+	+	−	+	+	−	+	+	−	+	+	−	+	+
$n\text{-}C_4H_9-\overset{\overset{O}{\|}}{S}-CH=CH-COOCH_3$		−	−	−	−	−	−	−	−	−	−	−	−	−	+	+
$n\text{-}C_5H_{11}-\overset{\overset{O}{\|}}{S}-CH=CH-COOCH_3$		−	−	−	−	−	−	−	+	+	−	−	−	±	+	+
$n\text{-}C_6H_{13}-\overset{\overset{O}{\|}}{S}-CH=CH-COOCH_3$		−	−	−	−	−	−	−	+	+	−	−	−	±	+	+
$n\text{-}C_8H_{17}-\overset{\overset{O}{\|}}{S}-CH=CH-COOCH_3$		−	−	−	−	−	−	±	+	+	−	+	+	+	+	+
$n\text{-}C_{10}H_{21}-\overset{\overset{O}{\|}}{S}-CH=CH-COOCH_3$		−	−	−	−	−	−	±	+	+	−	+	+	+	+	+

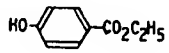
Compounds of the Invention	Organism Species	Staphylococcus aureus			Bacillus subtilis			Escherichia coli			Proteus vulgaris			Pseudomonas aeruginosa		
	Concentration of compounds (PPM)	500	100	50	500	100	50	500	100	50	500	100	50	500	100	50
$n\text{-}C_{12}H_{25}\text{-}S(=O)\text{-}CH{=}CH\text{-}COOCH_3$		–	–	–	–	–	–	–	±	+	±	+	+	+	+	+
$n\text{-}C_{14}H_{29}\text{-}S(=O)\text{-}CH{=}CH\text{-}COOCH_3$		–	–	–	–	–	–	+	+	+	+	+	+	+	+	+
$n\text{-}C_{16}H_{33}\text{-}S(=O)\text{-}CH{=}CH\text{-}COOCH_3$		–	–	–	–	–	–	+	+	+	+	+	+	+	+	+
$n\text{-}C_{18}H_{37}\text{-}S(=O)\text{-}CH{=}CH\text{-}COOCH_3$		–	–	±	–	–	+	+	+	+	+	+	+	+	+	+
 Reference Compound		+	+	+	+	+	+	+	+	+	+	+	+	+	+	+
 Reference Compound		–	+	+	±	+	+	±	+	+	–	+	+	+	+	+

Compounds of the Invention	Organism Species	Staphylococcus aureus			Bacillus subtilis			Escherichia coli			Proteus vulgaris			Pseudomonas aeruginosa		
	Concentration of compounds (PPM)	500	100	50	500	100	50	500	100	50	500	100	50	500	100	50
$n\text{-}C_4H_9\text{–}\overset{\overset{O}{\|}}{S}\text{–}CH{=}CH\text{–}COOH$		–	+	+	–	+	+	+	+	+	+	+	+	+	+	+
$n\text{-}C_4H_5\text{–}\overset{\overset{O}{\|}}{S}\text{–}CH{=}CH\text{–}COONa$		–	+	+	–	+	+	+	+	+	+	+	+	+	+	+
$n\text{-}C_4H_9\text{–}\overset{\overset{O}{\|}}{S}\text{–}CH{=}CH\text{–}COOCH_3$		–	–	–	–	–	–	–	–	–	–	–	–	–	+	+
$n\text{-}C_4H_9\text{–}\overset{\overset{O}{\|}}{S}\text{–}CH{=}CH\ COO(CH_2CH_2O)_2CH_3$		–	–	–	–	–	–	–	+	+	–	–	+	+	+	+
$n\text{-}C_4H_9\text{–}\overset{\overset{O}{\|}}{S}\text{–}CH{=}CH\text{–}COOCH_2\overset{\overset{OH}{\|}}{C}HCH_2OH$		–	–	–	–	–	–	–	–	+	–	–	+	±	+	+
		–	–	–	–	–	–	–	±	+	–	±	+	+	+	+
$n\text{-}C_4H_9\text{–}\overset{\overset{O}{\|}}{S}\text{–}CH{=}CH\text{–}COO(CH_2CH_2O)_{10}H$		–	–	–	–	–	–	–	–	+	–	±	+	±	+	+

Compounds of the Invention	Organism Species	Staphylococcus aureus			Bacillus subtilis			Escherichia coli			Proteus vulgaris			Pseudomonas aeruginosa		
	Concentration of compounds (PPM)	500	100	50	500	100	50	500	100	50	500	100	50	500	100	50
		–	–	–	–	–	–	–	+	+	–	+	+	+	+	+
		+	+	+	+	+	+	+	+	+	+	+	+	+	+	+
Reference Compound																
		–	+	+	±	+	+	±	+	+	–	+	+	+	+	+
Reference Compound																

Compounds	Organism Species:	Staphylococcus aureus			Bacillus subtilis			Escherichia coli			Proteus vulgaris			Pseudomonas aeruginosa		
	Concentration of compound (ppm)	1000	500	100	1000	500	100	1000	500	100	1000	500	100	1000	500	100
$n\text{-}C_4H_9\text{-}S(=O)\text{-}CH{=}CH\text{-}CON(CH_2CH_2OH)_2$		–	–	–	–	–	–	–	±	+	–	–	+	–	+	+
$n\text{-}C_6H_{13}\text{-}S(=O)\text{-}CH{=}CHCON(CH_2CH_2OH)_2$		–	–	–	–	–	±	–	+	+	–	+	+	+	+	+
$n\text{-}C_8H_{17}\text{-}S(=O)\text{-}CH{=}CHCON(CH_2CH_2OH)_2$		–	–	–	–	–	+	–	+	+	±	+	+	+	+	+
$n\text{-}C_{10}H_{21}\text{-}S(=O)\text{-}CH{=}CH\text{-}CON(CH_2CH_2OH)_2$		–	–	–	–	+	+	±	+	+	+	+	+	+	+	+
$n\text{-}C_{12}H_{23}\text{-}S(=O)\text{-}CH{=}CHCON(CH_2CH_2OH)_2$		–	–	–	–	–	–	–	+	+	–	+	+	±	+	+
$n\text{-}C_{14}H_{29}\text{-}S(=O)\text{-}CH{=}CHCON(CH_2CH_2OH)_2$		–	–	+	–	–	+	+	+	+	+	+	+	+	+	+
$n\text{-}C_{16}H_{33}\text{-}S(=O)\text{-}CH{=}CHCON(CH_2CH_2OH)_2$		–	+	+	–	+	+	+	+	+	+	+	+	+	+	+
$n\text{-}C_4H_9\text{-}S(=O)\text{-}CH{=}CH\text{-}CONH_2$		–	–	+	–	±	+	±	+	+	±	+	+	+	+	+
$n\text{-}C_4H_9\text{-}S(=O)\text{-}CH{=}CH\text{-}CONH_2$		–	–	–	–	–	–	–	±	+	–	±	+	–	+	+

Compounds	Organism Species:	Staphylococcus aureus			Bacillus subtilis			Escherichia coli			Proteus vulgaris			Pseudomonas aeruginosa		
	Concentration of compound (ppm)	1000	500	100	1000	500	100	1000	500	100	1000	500	100	1000	500	100
		–	–	+	–	–	+	–	+	+	–	+	+	±	+	+
		–	–	–	–	–	–	–	+	+	–	–	±	–	+	+
		–	–	–	–	–	–	–	±	+	–	–	+	–	+	+
		–	–	–	–	–	+	–	+	+	±	+	+	+	+	+
		–	–	+	–	–	+	+	+	+	+	+	+	+	+	+
$n\text{-}C_{11}H_{23}CON(CH_2CH_2OH)_2$ (Reference Compound)		–	+	+	–	±	+	+	+	+	+	+	+	+	+	+
 (Reference Compound)		–	+	+	–	+	+	±	+	+	±	+	+	+	+	+

Compounds	Organism Species:	Staphylococcus aureus			Bacillus subtilis			Escherichia coli			Proteus vulgaris			Pseudomonas aeruginosa		
	Concentration of compound (ppm)	1000	500	100	1000	500	100	1000	500	100	1000	500	100	1000	500	100
 (Reference Compound)		−	−	+	−	±	+	−	±	+	−	−	+	±	+	+

Example 7.

This Example shows the test results of the growth preventing effects of various compounds according to the present invention against fungi and yeasts.

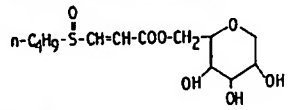
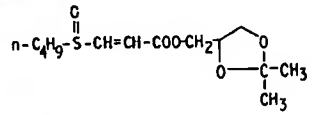
1 ml of a solution of each of the compounds having a predetermined concentration as set forth in the following Table and 19 ml of Sabouraud's agar culture medium preliminarily heated to a molten state were put into a Petri dish of 9 cm in diameter which had been sterilized in an autoclave, and uniformly mixed with each other and then solidified to form a plate.

In accordance with the general procedure of the "Method of Testing Resistance against Moulding" prescribed in Japanese Industrial Standard (JIS) No. Z—2911, on each of the Sabouraud's culture media individually containing specific compounds set forth in the Table below, there was coated one platinum loop of a suspension containing spores of Penicillium critrinum, Aspergillus niger or Trichophyton mentagrophytes or a suspension of Candida albicans which is a representative yeast. The organisms on the cultivation plates were cultivated for 5 days in a thermostatic chamber maintained at 25°C. After the end of the cultivation period, the growth states of the fungi and yeasts were observed, and the minimum concentrations of the respective compounds necessary for preventing growth of each organism were obtained to determine the effects of the compounds.

Compounds of the Invention	Organism Species	Penicillium citrinum			Aspergillus niger			Trichophyton mentagrophytes			Candida albicans		
	Concentration of compounds (PPM)	500	100	50	500	100	50	500	100	50	500	100	50
$CH_3-\overset{O}{\overset{\|}{S}}-CH=CH-COOCH_3$		+	+	+	+	+	+	−	±	+	±	+	+
$n\text{-}C_2H_5-\overset{O}{\overset{\|}{S}}-CH=CH-COOCH_3$		−	+	+	±	+	+	−	−	+	−	+	+
$n\text{-}C_3H_7-\overset{O}{\overset{\|}{S}}-CH=CH-COOCH_3$		−	+	+	−	+	+	−	−	+	−	±	+
$n\text{-}C_4H_9-\overset{O}{\overset{\|}{S}}-CH=CH-COOCH_3$		−	+	+	−	+	+	−	−	+	+	+	+
$n\text{-}C_5H_{11}-\overset{O}{\overset{\|}{S}}-CH=CH-COOCH_3$		+	+	+	−	+	+	−	−	+	+	+	+
$n\text{-}C_6H_{13}-\overset{O}{\overset{\|}{S}}-CH=CH-COOCH_3$		−	−	+	−	+	+	−	−	−	−	±	+
$n\text{-}C_8H_{17}-\overset{O}{\overset{\|}{S}}-CH=CH-COOCH_3$		−	−	−	−	+	+	−	−	−	−	−	−
$n\text{-}C_{10}H_{21}-\overset{O}{\overset{\|}{S}}-CH=CH-COOCH_3$		−	−	+	±	+	+	−	−	±	−	−	−

Compounds of the Invention	Organism Species	Penicillium citrinum			Aspergillus niger			Trichophyton mentagrophytes			Candida albicans		
	Concentration of compounds (PPM)	500	100	50	500	100	50	500	100	50	500	100	50
$n\text{-}C_{12}H_{25}-\overset{\overset{O}{\|}}{S}-CH=CH-COOCH_3$		+	+	+	+	+	+	−	−	+	−	−	−
$n\text{-}C_{14}H_{29}-\overset{\overset{O}{\|}}{S}-CH=CH-COOCH_3$		+	+	+	+	+	+	−	±	+	−	−	±
$n\text{-}C_{16}H_{33}-\overset{\overset{O}{\|}}{S}-CH=CH-COOCH_3$		+	+	+	+	+	+	−	+	+	−	+	+
$n\text{-}C_{18}H_{37}-\overset{\overset{O}{\|}}{S}-CH=CH-COOCH_3$		+	+	+	+	+	+	−	+	+	+	+	+
Potassium sorbate (Reference Compound)		+	+	+	+	+	+	−	+	+	+	+	+
Anhydrous sodium acetate (Reference Compound)		−	+	+	+	+	+	−	+	+	−	+	+

Compounds of the Invention	Organism Species	Penicillium citrinum			Aspergillus niger			Trichophyton mentagrophytes			Candida albicans		
	Concentration of compounds (PPM)	1000	500	100	1000	500	100	1000	500	100	1000	500	100
Potassium sorbate (Reference Compound)		±	+	+	±	+	+	−	−	+	+	+	+
Anhydrous sodium acetate (Reference Compound)		−	−	+	−	+	+	−	−	+	−	−	+

Compounds of the Invention	Organism Species	Penicillium citrinum			Aspergillus niger			Trichophyton mentagrophytes			Candida albicans		
	Concentration of compounds (PPM)	500	100	50	500	100	50	500	100	50	500	100	50
		+	+	+	+	+	+	−	+	+	−	±	+
		−	+	+	−	+	+	−	−	−	−	−	−
		−	+	+	−	−	+	−	−	−	−	±	+
		−	−	+	−	−	+	−	−	−	−	+	+
		−	+	+	−	+	+	−	−	+	+	+	+
		±	+	+	−	+	+	−	+	+	+	+	+
		−	+	+	−	+	+	−	−	+	−	±	+

Compounds of the Invention	Organism Species	Penicillium citrinum			Aspergillus niger			Trichophyton mentagrophytes			Candida albicans		
	Concentration of compounds (PPM)	500	100	50	500	100	50	500	100	50	500	100	50
Potassium sorbate (Reference Compound)		+	+	+	+	+	+	–	+	+	+	+	+
Anhydrous sodium acetate (Reference Compound)		–	+	+	+	+	+	–	+	+	–	+	+

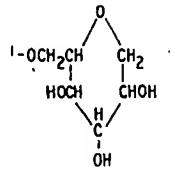
The compounds of the invention possess antimicrobial activity against one or more of bacteria, fungi and yeasts. They can be used as preservatives and antiseptics in cosmetic oils, lotions and creams, and pharmaceutical oil, lotion and cream compositions for topical application. They can be used in place of the preservatives and antiseptics conventionally used in such compositions, at approximately the same concentration levels. In particular, they can be used in place of sodium salicylate, ethyl paraben, potassium sorbate and anhydrous sodium acetate, which are conventional preservatives, in prior art compositions that employ those conventional preservatives.

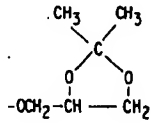
WHAT WE CLAIM IS:—

1. A compound having the formula:

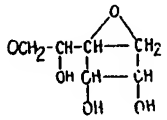
$$RSO—CH=CHX$$

wherein R is alkyl or alkenyl having 1 to 20 carbon atoms, or a naryl group, and X is —COY, wherein Y is (1) $—O(CH_2CH_2O)_mH$, wherein m is zero or an integer from 1 to 12, or (2) —OM, wherein M is an alkali metal, an alkaline earth metal or NH_4, or (3) $—O(CH_2CH_2O)_mR_1$, wherein m is as defined above and R_1 is alkyl having one to 20 carbon atoms, or (4) a hydroxyl-substituted alkoxy group obtained by removing one hydrogen atom from one hydroxyl group of a polyhydric aliphatic alcohol or a group of the formula





or

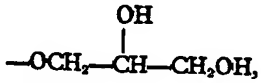


or (5) —NR'R", wherein R' is selected from hydrogen, alkyl having 1 to 20 carbon atoms, and hydroxyalkyl having 2 to 6 carbon atoms, and R" is selected from hydrogen, alkyl having 1 to 20 carbon atoms, and substituted alkyl having 2 to 6 carbon atoms wherein the substituent is selected from hydroxyl and a sulfo group in the form of a salt (—SO_3M_1, wherein M_1 is an alkali metal).

2. A compound according to Claim 1, wherein R is a straight-chain alkyl or alkenyl group having 3 to 18 carbon atoms.

3. A compound according to Claim 1, wherein Y is a hydroxyl group or OM where M is an alkali metal.

4. A compound according to Claim 1 or Claim 2, wherein Y is selected from alkoxy having one to 3 carbon atoms, alkoxyethoxy having one to 3 carbon atoms in the alkyl moiety, and —$O(CH_2CH_2O)_2R'$, where R' is C_1 to C_3 alkyl,



and —$O(CH_2CH_2O)_mH$ (m=1 to 12).

5. A compound according to Claim 1 or Claim 2, wherein Y is —NR'NR".

6. A compound according to Claim 5, wherein R' is selected from hydrogen, alkyl having 1 to 3 carbon atoms, and hydroxyalkyl group having 2 or 3 carbon atoms, and R" is selected from hydrogen, alkyl having 1 to 3 carbon atoms, hydroxyalkyl having 2 or 3 carbon atoms, and substituted alkyl group having 2 or 3 carbon atoms and wherein the substituent is —SO_3M wherein M is an alkali metal.

7. A method of preparing a compound as claimed in Claim 1, which comprises oxidizing a starting compound having the formula:

wherein X is as defined in claim 1, with an inorganic or organic peroxide.

8. A method according to Claim 7, wherein said inorganic or organic peroxide is hydrogen peroxide, sodium metaperiodate, *m*-chloro-perbenzoic acid, perbenzoic acid, or peracetic acid.

9. A method according to Claim 8, wherein 1.1 to 1.5 moles of said inorganic or organic peroxide is used per 1 mole of said starting compound.

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